

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF:

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DOCKET NO.: 3226-01

CUSTOMER NUMBER: 26645

SERIAL NO.: 10/554,481

EXAMINER: T. OLADAPO

FILED: OCTOBER 24, 2005

GROUP ART UNIT: 1797

TITLE: DIESEL LUBRICANT LOW IN SULFUR AND PHOSPHORUS
Wickliffe, Ohio

Hon. Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 C.F.R. §1.132

I, Virginia A. Carrick, declare as follows:

I received a bachelor of science degree with a major in chemistry in 1986 from John Carroll University. I have been employed by The Lubrizol Corporation since 1987 as a chemist. Since 1992 I have been responsible for formulating lubricants for various engines including stationary gas, heavy duty diesel, passenger car, compressed natural gas, and 4 stroke motorcycle engines. I am one of the inventors in the above-mentioned application, and I am familiar with the references which were used in the rejection thereof.

In order to further illustrate the improvement in performance of the compositions of the above invention, the following experiments were performed under my direction:

A series of lubricant formulations were prepared according to the teachings of the Nakazato et al. reference, JP 2002-053888 (English language machine translation). In particular, each formulation contained a set of common lubricant additives typical of

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I hereby certify that this Declaration is being filed electronically via the USPTO EFS to the Commissioner for Patents, United States Patent & Trademark Office, in accordance with §1.6(a)(4) on:

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an engine lubricant. These are set forth in the table, below. Additionally, each lubricant formulation contained either 0.3% or 0.5% by weight of an auxiliary antioxidant/antiwear agent as disclosed in paragraph 0039 of Nakazato. Two materials were selected from Nakazato which are not within the scope of the present claims: (a) oleyl amide, and (b) an amine phosphate salt (from 2-ethylhexyl amine, isoctyl alcohol, and phosphorus pentoxide). Two sulfurized olefin materials were chosen for testing which are within the scope of the present claims: (c) a dialkyl polysulfide prepared from isobutylene and S_2Cl_2/Na_2S , and (d) a sulfurized 4-carbutoxy cyclohexene.

Each lubricant formulation was subjected to an HFRR Ramp Wear test, consisting of a reciprocating ball on flat geometry, lubricated with the test fluid, test duration 75 minutes, 1 mm stroke, 20 Hz, temperature 40 °C for 15 minutes, then ramped to 160 °C at 2 °C/minute. Results are reported as wear scar diameter on the ball, μm . Lower numbers are better. Each lubricant was also subjected to two oxidation tests. The first is designated OXDN 200C. A specified amount of oil in a covered and vented test tube is blow with air under high temperature conditions (200 °C). After 24 hours, the viscosity of the oil is determined and compared with the viscosity at the start of the test. Lower % viscosity increase is better. The second oxidation test was a pressure differential scanning calorimeter test. The onset of oxidation activity is measured from the heat flow thermograph using a pressure differential scanning calorimeter supplied with pressurized air for the oxidative atmosphere and temperature controlled to 210 °C. Longer oxidation induction time (OIT) is better.

Formulations and Results are shown in the following table:

Example:	1*	2*	3	4	5*	6*	7	8
(a) Oleyl amide, %	0.3				0.5			
(b) Amine phosphate salt		0.3				0.5		
(c) Dialkyl polysulfide, %			0.3				0.5	
(d) S'd carbobutoxy cyclohexene, %				0.3				0.5
Other components, %:								
Viscosity modifier, olefin copolymer (90% oil)	8.2	8.2	8.2	8.2	8.2	8.2	8.2	8.2
Succinimide dispersant (50% oil)	7.2	7.2	7.2	7.2	7.2	7.2	7.2	7.2
Overbased Ca detergents (~43% oil)	2.43	2.43	2.43	2.43	2.43	2.43	2.43	2.43
Overbased Mg detergent (50% oil)	1.31	1.31	1.31	1.31	1.31	1.31	1.31	1.31
Antioxidants: phenolic + amine	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
Zinc dialkyldithiophosphate (9% oil)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Pour point depressant, ester polymer (54% oil)	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Thiadiazole inhibitor	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Commercial foam inhibitor	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Mineral Oil	bal. ^a	bal. ^a	bal. ^a	bal. ^a	bal. ^a	bal. ^a	bal. ^a	bal. ^a
Test Results								
HFR wear scar, μm	181	184	162	157	243	192	182	142
OXDN 200C								
Initial viscosity	106.8	107.2	108.2	108.5	106.7	107.0	108.4	107.8
End of test viscosity	171.7	191.3	165.7	164.1	186.8	198.5	161.6	152.6
% viscosity increase	60.8	78.5	53.1	51.2	75.1	85.5	49.1	41.6
PDSC, OIT (min)	64.2	60.3	61.3	75.5	61.9	56.6	58.6	73.3

* A comparative example

a Balance to = 100%

The results show that, in the HFRR wear test, both formulations containing the sulfurized olefins exhibited significantly improved antiwear performance compared with the comparative formulations, both at 0.3% and 0.5% treat rate. In the OXDN 200C test, both formulations containing the sulfurized olefins exhibited significantly improved antioxidancy performance than the comparative formulations, at both concentrations. (The PDSC test also showed improved oxidative performance for the sulfurized carbobutoxy cyclohexene at both concentrations, although the improvement was not observed for the dialkyl polysulfide. It is my understanding that the OXDN 200C test provides a more severe and realistic test of antioxidation performance than does the PDSC test.)

I further declare that all statements herein made of my own knowledge are true and all statements herein made on information and belief are believed to be true. I understand that willful false statements and the like are punishable by fine or imprisonment or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.



Virginia A. Carrick

06/25/10 (date)